659

REPORT ON STANDARDIZATION OF MICROCHEMICAL METHODS

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This report gives the recommendations of the Associate Referees and the Referee, based on a collaborative study of micromethods for analysis for sulfur, nitrogen, bromine, and chlorine in organic compounds. The studies were designed to determine which of the methods described in the literature and in common use would be suitable, either in its present form or in some modified form, as the official method. The collaborators submitted not only the analytical results but also detailed descriptions of the methods used. From a statistical analysis of these data for accuracy and precision and a correlation of the analytical details, it is recommended†—

(1) That since the Carius and catalytic combustion procedures are equaly precise and accurate they should be studied further so that an official method can be developed for sulfur.

(2) That the catalytic combustion method for sulfur include oxygen flow rates greater than 11 ml per minute and that the long furnace be maintained at temperatures above 750°C.

(3) That the combustion tubes used in the Carius method for sulfur analysis have a volume less than 12.5 ml.

(4) That either the titrimetric or the gravimetric procedure be used for the above mentioned sulfur methods, since they are equally accurate.

(5) That further modification of the Kjeldahl method and of the pretreatment of samples be made before a method be recommended for the analysis of nitrogen in compounds containing the N-N linkage.

(6) That the method of pretreatment of samples prior to Kjeldahl nitrogen analysis used this year be further tested for compounds containing NO and NO_2 groups.

(7) Since the Pregl and Carius methods for analysis of bromine and chlorine in organic compounds are equally precise and accurate, that these two methods be studied further to develop an official method for analysis of these halogens.

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† For report of Subcommittee C and action of the Association, see *This Journal*, 35, 53 (1952).